

The Application of SW-846 Method 8261 to Analytes Required in Superfund's Current Statement of Work, SOM01.1. Part Two; Method Performance for Water

Michael H. Hiatt

U.S. Environmental Protection Agency

National Exposure Research Laboratory

Environmental Sciences Division

P.O. Box 93478, Las Vegas, Nevada 89193-3478

Introduction

Method 8261 provides analysis of problematic matrices, introducing novel quality tools including the determination of method performance by analyte in each analysis. This method uses analyte chemical properties, boiling point, and relative volatilities to measure method performance as functions of those chemical properties. Boiling point values for compounds are readily available in the literature but relative volatility values are unique to Method 8261. An overview of method 8261 chemistry is available on these web pages (Ref 1) (<http://www.epa.gov/nerlesd1/chemistry/vacuum/training/pdf/theory-rev5.pdf>).

All of the volatile organic analytes listed in Superfund's Contract Laboratory Program (CLP) SOM01.1 (ref 2) had not been evaluated using method 8261(Ref 3). In addition the CLP has a trace-level protocol for reporting trace-level contaminants in water using 25 mL samples. This study was also performed to provide initial data demonstrating the viability of the additional compounds and the application of the method for trace analyses.

Experimental

Vacuum Distiller. A Cincinnati Analytical Instruments vacuum distiller (Model VDC1012) performed the distillations in the study. The operating conditions are presented in Table 1. Vacuum distillation times were varied, and the flush time was extended so that the vacuum distillation cycle time matched the GC/MS cycle time.

Table 1. Vacuum Distillation Parameters	
<i>Stage</i>	Time (min)
Vacuum distillation	7.5
Transfer	2.5
Bake-out	2.5
<i>Temperature</i>	°C
Condenser heat	95
Condenser cool	-10
Cryotrap desorb	120
Cryotrap trapping	-160
Cryotrap bake-out	200
Transfer line	200
Vacuum distiller lines	95
Multiport valve	200
Autosampler lines	95
<i>Decontamination</i>	
Cycles	16
Pressurization time (min)	0.1
Evacuation time (min)	1.4

GC/MS Apparatus. The vacuum distiller is interfaced to a GC/MS so that the vacuum distillate is transferred directly to the GC/MS for analysis after a distillation. In this study, the GC/MS was a Thermo DSQ mass spectrometer and Trace GC. The GC capillary column was a 30 m x 0.25 mm i.d., 1.5 μ m film VOCOL column (Supelco, Bellefonte, PA). The GC operating conditions were 2.5 min at -20 °C, 40 °C/min ramp to 60 °C, 5 °C/min ramp to 120 °C and held at 120 °C for 1 min, 20 °C/min ramp to 220 °C and held for 12 min resulting in a GC run time of 34 min. The injection was split 30:1 with a constant flow rate of 1.4 ml/min. The mass spectrometer scanned between 35 and 300 amu at 1 scan/sec.

Study Approach. Standards containing all CLP analytes plus Method 8261 analytes were prepared at varying levels to identify the limit of quantitation as well as identify any potential interference. Triplicate analyses of 5 mL water and 25 mL water samples, spiked with mid-level and low-level amounts of analytes were used to document method performance. Data processing that is unique to Method 8261 was performed by the software available on the web (ref 4).

Results and Discussion

The calibration data in this document was also used in a presentation “Creating the Calibration Curve and Generating Method 8261 Quantitation Reports through SMCRReporter V4.0” (Ref 5) (<http://www.epa.gov/nerlesd1/chemistry/vacuum/methods/software.htm#calibration>). The calibration curve can be downloaded (and data that was used to generate it) along with the presentation. The calibration ranges demonstrated in this study for each analyte are presented in Table 2.

Note that some of the compounds’ lower standards were raised due to background interference. Some compounds also are detected in blanks and are likely due to proximity of common laboratory solvents. Naphthalene and methyl naphthalene are due to persistent low amounts in system blanks.

A few of the analytes had spectral interferences that made low-mass sensitivity poor so that their lower standards data points were not used, elevating the LOQ to the levels noted in Table 2. Propionitrile was the only compound that had a calibration error greater than 25 % (29%) and tends to have interference that makes integration unreliable.

A unique attribute of Method 8261 in that calibration is in units of mass, and not concentration. Because of Method 8261’s use of internal standards to mitigate the effects from matrices (like water), the method is not as dependent on sample size or matrix as other methods. Therefore the low mass in the calibration curve is used as the limit of quantitation. Therefore, for method 8261 the limit of quantitation (LOQ) is determined as the low standard divided by the sample size.

Therefore, we can look at the low calibration point in the calibration curve and determine the LOQ by simply dividing by the sample size desired. For instance, the low standard value of 1.5 ng divided by 5 mL results in a LOQ of 0.3 ng/mL.

The LOQ values determined in this study are consistently lower than the CLP requirement for low-level water (5 mL) and trace-level water (25 mL) samples. In fact, the LOQ (2 µg/L) for 1,4-dioxane found for 25-mL water samples using Method 8261 met the CLP selected ion monitoring method (2 µg/L). The LOQ (0.06 µg/L) for 1,2-dibromoethane and 1,2-dibromo-3-chloropropane in 25-mL water samples using Method 8261 nearly met the CLP selected ion monitoring method (0.05 µg/L). The LOQ for 1,2-dibromoethane and 1,2-dibromo-3-chloropropane may have easily been lowered with selection of a slightly lower calibration point during calibration, but this had not been an objective of this study.

Table 2. Analytes

Volatile Organic Compounds	High Standard (ng) ^a	Low Standard (ng) ^b	LOQ 5 mL (µg/L)	LOQ 25 mL (µg/L)	CLP 5 mL ^c (µg/L)	CLP 25 mL ^c (µg/L)
Dichlorodifluoromethane	500	1.5	0.3	0.06	5	0.5
Chloromethane	500	1.5	0.3	0.06	5	0.5
Vinyl chloride	500	1.5	0.3	0.06	5	0.5
Bromomethane	500	1.5	0.3	0.06	5	0.5
Chloroethane	500	5.0 ^d	1.0	0.2	5	0.5
Trichlorofluoromethane	500	1.5	0.3	0.06	5	0.5
Diethyl ether	1000	10 ^d	2.0	0.40		
1,1,2-Trichloro-1,2,2-trifluoroethane	500	1.5	0.3	0.06	5	0.5
Acetone	1000	1000 ^e	200 ^e	40 ^e	10	5
1,1-Dichloroethene	500	5 ^d	1.0	0.20	5	0.5
Iodomethane	1000	3	0.6	0.12		
Allyl chloride	500	5 ^c	1.	0.2		
Acetonitrile	2000	6	1.2	0.24		
Methyl acetate	500	5 ^d	1.	0.2	5	0.5
Carbon disulfide	500	1.5	0.3	0.06	10	5
Methylene chloride	500	25 ^e	5. ^e	1. ^e	5	0.5
MTBE	500	1.5	0.3	0.06	5	0.5
Acrylonitrile	1000	3	0.6	0.12		
<i>trans</i> -1,2-Dichloroethene	500	1.5	0.3	0.06	5	0.5
1,1-Dichloroethane	500	1.5	0.3	0.06	5	0.5
2,2-Dichloropropane	500	1.5	0.3	0.06		
Propionitrile	1000	10	2	0.4		
2-Butanone	2000	100 ^e	20 ^e	4 ^e	10	5
<i>cis</i> -1,2-Dichloroethene	500	1.5	0.3	0.06	5	0.5
Methacrylonitrile	1000	10 ^d	2	0.4		

Chloroform	500	1.5	0.3	0.06	5	0.5
Bromochloromethane	500	1.5	0.3	0.06	5	0.5
Cyclohexane	500	1.5	0.3	0.06	5	0.5
1,1,1-Trichloroethane	500	1.5	0.3	0.06	5	0.5
1,1-Dichloropropene	500	1.5	0.3	0.06		
Carbon tetrachloride	500	1.5	0.3	0.06	5	0.5
1,2-Dichloroethane	500	5 ^d	1.	0.2	5	0.5
Benzene	500	1.5	0.3	0.06	5	0.5
Trichloroethene	500	1.5	0.3	0.06	5	0.5
Methyl cyclohexane	500	1.5	0.3	0.06	5	0.5
1,2-Dichloropropane	500	1.5	0.3	0.06	5	0.5
Methyl methacrylate	1000	3	0.6	0.12		
Dibromomethane	500	1.5	0.3	0.06		
Bromodichloromethane	500	1.5	0.3	0.06	5	0.5
1,4-Dioxane	5000	50 ^f	10	2	100	20
4-Methyl-2-pentanone	2000	6	0.3	0.06	10	5
<i>trans</i> -1,3-Dichloropropene	2500	7.5	0.3	0.06	5	0.5
Toluene	500	1.5	0.3	0.06	5	0.5
Pyridine	7500	75 ^f	0.3	0.06		
<i>cis</i> -1,3-Dichloropropene	2500	7.5	0.3	0.06		
2-Hexanone	1002	3.0	0.6	0.12	10	5
1,1,2-Trichloroethane	500	1.5	0.3	0.06	5	0.5
1,3-Dichloropropane	500	1.5	0.3	0.06		
Tetrachloroethene	500	1.5	0.3	0.06	5	0.5
Dibromochloromethane	500	1.5	0.3	0.06	5	0.5
1,2-Dibromoethane	500	1.5	0.3	0.06	5	0.5
Chlorobenzene	500	1.5	0.3	0.06	5	0.5
1,1,1,2-Tetrachloroethane	500	1.5	0.3	0.06		
Ethylbenzene	500	1.5	0.3	0.06	5	0.5
<i>m,p</i> -Xylenes	500	1.5	0.3	0.06	5	0.5
<i>o</i> -Xylene	500	1.5	0.3	0.06	5	0.5
Styrene	500	1.5	0.3	0.06	5	0.5
Isopropylbenzene	500	1.5	0.3	0.06	5	0.5
bromoform	500	1.5	0.3	0.06	5	0.5
<i>cis</i> -1,4-Dichloro-2-butene	2000	6	0.3	0.06		
1,1,2,2-Tetrachloroethane	500	1.5	0.3	0.06	5	0.5
1,2,3-Trichloropropane	500	1.5	0.3	0.06		
Propylbenzene	500	1.5	0.3	0.06		
Bromobenzene	500	1.5	0.3	0.06		
<i>trans</i> -1,4-Dichloro-2-butene	2000	6	0.3	0.06		
1,3,5-Trimethylbenzene	500	1.5	0.3	0.06		
2-Chlorotoluene	500	1.5	0.3	0.06		
4-Chlorotoluene	500	1.5	0.3	0.06		
<i>tert</i> -Butylbenzene	500	1.5	0.3	0.06		
<i>sec</i> -Butylbenzene	500	1.5	0.3	0.06		
Pentachloroethane	500	1.5	0.3	0.06		

1,2,4-Trimethylbenzene	500	1.5	0.3	0.06		
<i>p</i> -Isopropyltoluene	500	1.5	0.3	0.06		
1,3-Dichlorobenzene	500	1.5	0.3	0.06	5	0.5
1,4-Dichlorobenzene	500	1.5	0.3	0.06	5	0.5
<i>n</i> -Butylbenzene	500	1.5	0.3	0.06		
1,2-Dichlorobenzene	500	1.5	0.3	0.06	5	0.5
Acetophenone	1000	50 ^d	10	2		
1,2-Dibromo-3-chloropropane	500	1.5	0.3	0.06	5	0.5
Nitrobenzene	1000	10 ^d	2	0.4		
1,2,4-Trichlorobenzene	500	1.5	0.3	0.06	5	0.5
Hexachlorobutadiene	500	1.5	0.3	0.06		
Naphthalene	500	5 ^e	1 ^e	0.2 ^e		
1,2,3-Trichlorobenzene	500	1.5	0.3	0.06	5	0.5
2-Methylnaphthalene	1000	10 ^e	2 ^e	0.4 ^e		
1-Methylnaphthalene	1000	10 ^e	2 ^e	0.4 ^e		

^a Five standard runs generated the curve. The high standard is listed and the other four runs as fractions of the high standard are 0.3, 0.05, 0.01, and .003.

^b Lowest viable standard is listed. Those points that are not 0.003 times the high standard are identified

^c The LOQ are from the CLP organics contract SOM01.1

^d Amount was raised to minimize bias of results from system contamination.

^e Spectral interference that made integration of lower standard data points unreliable

^f Memory effects that gave high bias to responses of lower data points making them unreliable.

The next issue was testing the method performance by running triplicate analyses of mid-level and low-level concentrations of 5 mL and 25 mL samples. The samples were quantitated using the calibration curve and the results are presented in tables 3 and 4.

With the exception of those analytes present in the reagent blanks, the method provides good method performance. There appeared to be no interference among most of the analytes.

However, the more polar compounds sometimes require manual integration depending on the level of interference.

Table 3. Sample Performance for 5-mL Samples

Volatile Organic Compounds	Mid-level spike (ng)	Recovery (%)	Precision (%)	Low-level spike (ng)	Recovery (%)	Precision (%)
Dichlorodifluoromethane	50	112.8	14.4	1	85.7	6.5
Chloromethane	50	109.2	7.3	1	99.7	2.5
Vinyl chloride	50	119.8	11.8	1	81.7	5.8
Bromomethane	50	100.4	5.2	1	87.7	9.0
Chloroethane	50	103.8	12.9	1	124.7	26.1
Trichlorofluoromethane	50	108.4	1.9	1	107.0	8.9
Diethyl ether	100	100.8	2.7	2	113.3	28.2
1,1,2-Trichloro-1,2,2-trifluoroethane	50	110.3	1.1	1	99.7	7.5

Acetone	100	93.7	4.8	2	1578.3	477.9
1,1-Dichloroethene	50	118.4	1.5	1	113.7	17.9
Iodomethane	100	105.7	2.3	2	106.3	18.5
Allyl chloride	50	90.7	1.8	1	106.0	37.3
Acetonitrile	200	102.2	1.5	4	112.8	23.8
Methyl acetate	50	80.2	0.9	1	121.7	21.5
Carbon disulfide	50	98.3	7.1	1	116.7	6.1
Methylene chloride	50	81.1	3.6	1	152.3	9.3
MTBE	50	110.2	1.0	1	102.7	8.4
Acrylonitrile	100	88.4	0.1	2	90.8	12.5
<i>trans</i> -1,2-Dichloroethene	50	116.1	1.5	1	97.3	4.7
1,1-Dichloroethane	50	107.1	1.4	1	97.0	6.0
2,2-Dichloropropane	50	97.9	0.8	1	92.7	7.1
Propionitrile	100	88.5	13.3	2	88.0	23.8
2-Butanone	200	68.6	6.4	4	147.7	15.2
<i>cis</i> -1,2-Dichloroethene	50	107.7	1.3	1	104.0	11.8
Methacrylonitrile	100	87.3	0.9	2	103.7	7.1
Chloroform	50	103.9	1.6	1	106.0	6.0
Bromochloromethane	50	111.7	1.1	1	95.0	6.1
Cyclohexane	50	114.1	0.8	1	106.0	2.6
1,1,1-Trichloroethane	50	124.5	0.6	1	110.7	5.8
1,1-Dichloropropene	50	109.9	1.8	1	91.0	9.8
Carbon tetrachloride	50	115.0	0.6	1	100.7	11.5
1,2-Dichloroethane	50	107.9	2.4	1	113.3	19.0
Benzene	50	102.8	0.9	1	106.0	6.2
Trichloroethene	50	114.9	1.0	1	90.0	34.1
Methyl cyclohexane	50	111.2	0.9	1	98.7	13.7
1,2-Dichloropropane	50	105.1	2.1	1	101.7	6.1
Methyl methacrylate	100	93.1	1.2	2	97.0	3.1
Dibromomethane	50	100.3	1.9	1	84.7	6.4
Bromodichloromethane	50	99.7	1.8	1	93.3	1.2
1,4-Dioxane	500	95.3	3.5	10	112.3	14.1
4-Methyl-2-pentanone	200	92.2	1.5	4	81.0	18.4
<i>trans</i> -1,3-Dichloropropene	250	105.3	1.5	5	103.4	4.2
Toluene	50	104.1	1.1	1	105.3	4.7
Pyridine	750	71.2	13.9	15	129.1	37.2
<i>cis</i> -1,3-Dichloropropene	250	104.5	2.3	5	100.1	1.6
2-Hexanone	100	97.9	2.0	2	83.7	6.3
1,1,2-Trichloroethane	50	109.6	1.6	1	98.7	1.5
1,3-Dichloropropane	50	105.0	0.8	1	96.0	7.0
Tetrachloroethene	50	117.7	1.5	1	103.7	4.6
Dibromochloromethane	50	109.4	1.7	1	87.0	2.0
1,2-Dibromoethane	50	100.6	0.4	1	95.3	7.6
Chlorobenzene	50	105.3	1.6	1	105.7	5.9
1,1,1,2-Tetrachloroethane	50	118.7	1.0	1	95.7	24.0
Ethylbenzene	50	109.6	1.7	1	103.7	0.6

<i>m,p</i> -Xylenes	50	108.0	1.7	1	104.0	3.6
<i>o</i> -Xylene	50	106.2	2.2	1	101.7	2.9
Styrene	50	105.0	1.8	1	95.7	7.4
Isopropylbenzene	50	114.9	1.8	1	101.3	0.6
bromoform	50	112.4	0.8	1	84.0	10.4
<i>cis</i> -1,4-Dichloro-2-butene	200	104.1	0.6	4	93.4	2.5
1,1,2,2-Tetrachloroethane	50	105.7	2.5	1	92.3	1.2
1,2,3-Trichloropropane	50	103.6	1.4	1	99.0	3.5
Propylbenzene	50	109.5	1.3	1	100.0	3.0
Bromobenzene	50	106.8	0.6	1	95.0	2.6
<i>trans</i> -1,4-Dichloro-2-butene	200	103.5	1.5	4	90.3	7.4
1,3,5-Trimethylbenzene	50	109.7	1.5	1	95.3	0.6
2-Chlorotoluene	50	105.1	0.9	1	102.7	1.5
4-Chlorotoluene	50	104.1	2.0	1	99.3	3.5
<i>tert</i> -Butylbenzene	50	115.7	3.8	1	100.0	4.4
<i>sec</i> -Butylbenzene	50	102.2	1.6	1	97.0	3.6
Pentachloroethane	50	122.4	4.7	1	90.7	7.5
1,2,4-Trimethylbenzene	50	118.3	2.1	1	104.7	2.5
<i>p</i> -Isopropyltoluene	50	110.2	3.2	1	103.7	0.6
1,3-Dichlorobenzene	50	100.5	2.0	1	100.3	1.5
1,4-Dichlorobenzene	50	98.8	1.6	1	103.7	2.3
<i>n</i> -Butylbenzene	50	108.0	2.2	1	99.3	4.5
1,2-Dichlorobenzene	50	101.9	2.2	1	114.0	3.6
Acetophenone	100	98.5	22.8	2	154.0	20.8
1,2-Dibromo-3-chloropropane	50	101.8	2.4	1	87.0	8.5
Nitrobenzene	100	80.5	9.9	2	82.8	16.3
1,2,4-Trichlorobenzene	50	90.9	2.7	1	107.7	4.0
Hexachlorobutadiene	50	110.9	12.1	1	104.3	12.3
Naphthalene	50	88.6	5.7	1	97.3	5.0
1,2,3-Trichlorobenzene	50	97.8	2.8	1	97.0	6.2
2-Methylnaphthalene	100	118.4	7.6	2	106.2	7.9
1-Methylnaphthalene	100	113.3	8.5	2	109.7	9.3

Table 4. Sample Performance for 25-mL Samples

Volatile Organic Compounds	Mid-level spike (ng/mL)	Recovery	Precision	Low-level spike (ng/mL)	Recovery	Precision
		(%)	(%)		(%)	(%)
Dichlorodifluoromethane	10	70.2	12.1	0.2	71.7	12.6
Chloromethane	10	82.9	6.1	0.2	80.0	0.0
Vinyl chloride	10	99.7	5.9	0.2	90.0	5.0

Bromomethane	10	91.0	3.9	0.2	95.0	14.1
Chloroethane	10	104.0	4.8	0.2	130.0	21.8
Trichlorofluoromethane	10	108.3	5.1	0.2	118.3	2.9
Diethyl ether	20	100.3	2.1	0.4	130.8	5.8
1,1,2-Trichloro-1,2,2-trifluoroethane	10	113.8	5.6	0.2	103.3	7.6
Acetone	20	132.6	7.7	0.4	4253.3	92.3
1,1-Dichloroethene	10	115.5	2.4	0.2	131.7	5.8
Iodomethane	20	103.8	2.4	0.4	121.7	7.6
Allyl chloride	10	85.9	3.0	0.2	140.0	30.0
Acetonitrile	40	87.1	2.9	0.8	100.4	14.4
Methyl acetate	10	87.6	1.4	0.2	136.7	20.2
Carbon disulfide	10	92.0	2.0	0.2	113.3	7.6
Methylene chloride	10	81.4	2.7	0.2	173.3	18.9
MTBE	10	106.5	2.1	0.2	111.7	2.9
Acrylonitrile	20	98.5	1.3	0.4	89.2	14.2
<i>trans</i> -1,2-Dichloroethene	10	118.6	1.8	0.2	115.0	5.0
1,1-Dichloroethane	10	109.8	1.0	0.2	105.0	5.0
2,2-Dichloropropane	10	92.6	2.0	0.2	100.0	8.7
Propionitrile	20	89.3	22.7	0.4	96.7	10.1
2-Butanone	40	71.9	11.3	0.8	211.7	17.1
<i>cis</i> -1,2-Dichloroethene	10	109.1	4.2	0.2	113.3	11.5
Methacrylonitrile	20	95.3	3.1	0.4	105.0	7.5
Chloroform	10	103.8	3.5	0.2	120.0	0.0
Bromochloromethane	10	111.5	4.1	0.2	106.7	14.4
Cyclohexane	10	116.3	4.6	0.2	113.3	5.8
1,1,1-Trichloroethane	10	124.8	1.8	0.2	121.7	14.4
1,1-Dichloropropene	10	108.0	1.0	0.2	103.3	10.4
Carbon tetrachloride	10	113.1	1.5	0.2	100.0	0.0
1,2-Dichloroethane	10	104.7	3.2	0.2	118.3	12.6
Benzene	10	104.3	1.0	0.2	115.0	5.0
Trichloroethene	10	113.3	0.4	0.2	111.7	5.8
Methyl cyclohexane	10	114.4	5.1	0.2	91.7	10.4
1,2-Dichloropropane	10	102.1	0.9	0.2	105.0	13.2
Methyl methacrylate	20	96.9	3.3	0.4	100.0	2.5
Dibromomethane	10	98.1	3.8	0.2	88.3	10.4
Bromodichloromethane	10	93.0	3.4	0.2	108.3	5.8
1,4-Dioxane	100	92.6	5.3	2	130.0	7.4
4-Methyl-2-pentanone	40	95.7	2.0	0.8	86.7	16.5
<i>trans</i> -1,3-Dichloropropene	50	102.2	3.0	1	110.3	7.2
Toluene	10	107.6	0.5	0.2	115.0	0.0
Pyridine	150	81.1	11.3	3	135.9	10.2
<i>cis</i> -1,3-Dichloropropene	50	100.5	2.8	1	103.0	1.0
2-Hexanone	20	96.2	2.1	0.4	74.0	10.1
1,1,2-Trichloroethane	10	107.3	2.4	0.2	106.7	7.6
1,3-Dichloropropane	10	103.4	2.2	0.2	105.0	5.0
Tetrachloroethene	10	122.0	3.9	0.2	120.0	10.0

Dibromochloromethane	10	105.3	4.1	0.2	100.0	8.7
1,2-Dibromoethane	10	102.7	0.4	0.2	110.0	5.0
Chlorobenzene	10	107.4	0.6	0.2	115.0	0.0
1,1,1,2-Tetrachloroethane	10	114.1	0.6	0.2	101.7	2.9
Ethylbenzene	10	119.7	4.1	0.2	115.0	5.0
<i>m,p</i> -Xylenes	10	115.5	3.5	0.2	113.3	2.9
<i>o</i> -Xylene	10	109.1	1.4	0.2	111.7	2.9
Styrene	10	105.7	0.8	0.2	110.0	5.0
Isopropylbenzene	10	123.0	5.8	0.2	108.3	2.9
bromoform	10	111.7	3.2	0.2	95.0	10.0
<i>cis</i> -1,4-Dichloro-2-butene	40	104.5	1.0	0.8	86.7	10.5
1,1,2,2-Tetrachloroethane	10	100.1	2.6	0.2	101.7	2.9
1,2,3-Trichloropropane	10	106.7	1.9	0.2	85.0	27.8
Propylbenzene	10	118.2	6.6	0.2	108.3	2.9
Bromobenzene	10	106.5	2.3	0.2	105.0	8.7
<i>trans</i> -1,4-Dichloro-2-butene	40	103.3	1.0	0.8	100.4	8.1
1,3,5-Trimethylbenzene	10	119.8	4.7	0.2	111.7	7.6
2-Chlorotoluene	10	111.5	2.4	0.2	111.7	2.9
4-Chlorotoluene	10	107.9	1.1	0.2	108.3	5.8
<i>tert</i> -Butylbenzene	10	130.3	8.5	0.2	113.3	11.5
<i>sec</i> -Butylbenzene	10	105.6	1.3	0.2	110.0	0.0
Pentachloroethane	10	114.6	1.9	0.2	100.0	5.0
1,2,4-Trimethylbenzene	10	137.9	12.7	0.2	113.3	2.9
<i>p</i> -Isopropyltoluene	10	129.2	6.1	0.2	120.0	5.0
1,3-Dichlorobenzene	10	103.2	0.8	0.2	111.7	5.8
1,4-Dichlorobenzene	10	102.3	2.1	0.2	108.3	7.6
<i>n</i> -Butylbenzene	10	124.4	4.9	0.2	110.0	5.0
1,2-Dichlorobenzene	10	103.1	2.7	0.2	120.0	5.0
Acetophenone	20	116.3	25.4	0.4	240.0	49.2
1,2-Dibromo-3-chloropropane	10	108.3	4.2	0.2	103.3	16.1
Nitrobenzene	20	94.8	7.7	0.4	120.0	11.5
1,2,4-Trichlorobenzene	10	88.8	2.2	0.2	105.0	10.0
Hexachlorobutadiene	10	135.9	7.7	0.2	120.0	13.2
Naphthalene	10	90.9	3.8	0.2	106.7	2.9
1,2,3-Trichlorobenzene	10	89.6	0.7	0.2	96.7	10.4
2-Methylnaphthalene	20	118.8	1.5	0.4	129.2	5.2
1-Methylnaphthalene	20	114.8	1.3	0.4	135.0	2.5

Conclusion

The use of Method 8261 to determine Superfund's list of volatile analytes easily meets or exceeds the CLP criteria for water. While the studies did not address soil samples, such matrices have been the forte of the method and CLP criteria of soils should be easily met as well.

References

1. Hiatt, M.H., "An Overview of SW-846 Method 8261 Chemistry" Presentation posted on the vacuum distillation web page May 2006.
<http://www.epa.gov/nerlesd1/chemistry/vacuum/training/pdf/theory-rev5.pdf>
2. Superfund Contract Laboratory Statement of Work, SOM01.1 posted
<http://www.epa.gov/superfund/programs/clp/som1.htm>
3. Hiatt, M. H. Anal Chem. 1995, 67, 4044-4052. EPA version is posted
<http://www.epa.gov/nerlesd1/chemistry/vacuum/reference/analysis/anal.htm>
4. SMCReporter Version 4.0
<http://www.epa.gov/nerlesd1/chemistry/vacuum/methods/software.htm>
5. Hiatt, M.H., "Creating the Calibration Curve and Generating Method 8261 Quantitation Reports through SMCReporter V4.0" Presentation posted on the vacuum distillation web page May 2006.
(<http://www.epa.gov/nerlesd1/chemistry/vacuum/methods/software.htm#calibration>)